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Indian Standard
METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

PART 5 SPECIAL TEST METHODS FOR POLYAMIDE RESINS

(*Second Revision*)

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PART 5 SPECIAL TEST METHODS FOR POLYAMIOE RESINS

(Second Revision)

(*Page* 3, *clause* **0.2**, *line* 7) — Delete the words 'chlorinated rubber'.

(*Page* 3, *clause* **0.2**, *last line*) — Delete 'Part 8'.

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Indian Standard

METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

PART 5 SPECIAL TEST METHODS FOR POLYAMIDE RESINS

(Second Revision)

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Indian Standard

METHODS OF SAMPLING AND TEST FOR RESINS FOR PAINTS

PART 5 SPECIAL TEST METHODS FOR POLYAMIDE RESINS

(Second Revision)

0. FOREWORD

0.1 This Indian Standard (Part 5) (Second Revision) was adopted by the Indian Standards Institution on 18 August 1986, after the draft finalized by the Raw Materials for Paint Industry Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard was originally published in 1952 covering methods of sampling and general test methods mainly for natural resins. Subsequently, an Indian Standard for methods of sampling and test for natural and synthetic resins was published as Part 2 of the above standard in 1971. These two parts were amalgamated and revised in 1976. This revision has been necessitated as more and more newer synthetic resins like polyamidcs, polyvinyls, chlorinated rubber and emulsion polymers are being manufactured and used in the country. While revising the standard, the Committee felt it appropriate to publish this standard in various parts, as indicated below:

Part 1	General test methods
Part 2	Special test methods for alkyd resins
Part 3	Special test methods for phenolic resins
Part 4	Special test methods for epoxy resins
Part 5	Special test methods for polyamide resins
Part 6	Special test methods for amino resins
Part 7	Special test methods for determination of monomer content in acrylic or vinylacetate containing polymers and emulsions
Part 8	Special test methods for chlorinated rubber

0.3 In this standard (Part 5), test methods for identification, determination of acid value, amine value for reactive polyamides and non-reactive polyamides have been specified.

0.4 In reporting the result of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 5) prescribes the special test methods for polyamide resins used in paints and enamels.

2. TERMINOLOGY

2.1 For the purpose of this standard (Part 5), the definitions given in IS : 1303-1983† and IS : 6667-1972‡ shall apply.

3. SAMPLING

3.1 Representative samples of polyamide resins shall be drawn as prescribed in **3** of Part 1 of this standard.

4. IDENTIFICATION

4.1 Non-reactive polyamides are red brown transparent solids, while reactive polyamides are yellowish viscous liquids with a faint amine odour.

5. DETERMINATION OF AMINE VALUE

5.0 Outline of the Method — The methods for reactive and non-reactive polyamides differ from each other, basically both are by titration against standard acid solutions.

5.1 Reactive Polyamides

5.1.1 *Reagents*

5.1.1.1 *Standard sodium acetate solution* — 0.1 N. Dissolve 5.3 g of sodium acetate (anhydrous) in glacial acetic acid and dilute to 100 ml.

5.1.1.2 *Crystal voilet indicator solution* — Dissolve 1.25 g of crystal violet in 250 ml of glacial acid.

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*Rules for rounding off numerical values (*revised*).

†Glossary of terms relating to paints (*second revision*).

‡Glossary of terms used in synthetic resin industry.

5.1.1.3 *Standard perchloric acid solution* — 0.1 N. Dissolve 5.5 ml of concentrated perchloric acid, 7.5 ml acetic anhydride and dilute to 500 ml with glacial acetic acid. Standardize the solution against standard sodium acetate solution using crystal violet indicator. End point is blue to green.

5.1.2 *Procedure* — Weigh 0.3 to 0.4 g of resin sample in a conical flask. Add 50 ml glacial acetic acid and dissolve the resin in the same. Add 2-3 drops of crystal violet indicator and titrate against standard perchloric acid solution. End point is blue to green. Express the result in milligrams of potassium hydroxide required per gram of sample.

5.1.3 *Calculation*

$$\text{Amine value} — \frac{V \times N \times 56\cdot 1}{M}$$

where

V = volume in ml of standard perchloric acid solution used,

N = normality of standard perchloric acid solution, and

M = mass in g of sample of resin taken for the test.

5.2 Non-reactive Polyamides

5.2.1 *Reagents*

5.2.1.1 *Isopropyl alcohol*

5.2.1.2 *Bromophenol blue indicator* — 1 percent.

5.2.1.3 *Standard hydrochloric acid solution* — 0.1 N. Dissolve 17 ml of concentrated hydrochloric acid (sp gr 1.19) in *iso*propyl alcohol and dilute to 2 litres. Standardize this solution against standard alcoholic potassium hydroxide solution using bromophenol blue as indicator.

5.2.2 *Procedure*

5.2.2.1 Dissolve 3-4 g of resin in 25-30 ml *iso*propyl alcohol, in a conical flask, add a few drops of bromophenol indicator and titrate against standard hydrochloric acid solution. End point is greenish blue to lemon yellow. Express the result in milligrams of potassium hydroxide required per gram of sample.

5.2.3 *Calculation*

$$\text{Amine value} = \frac{V \times N \times 56\cdot 1}{M}$$

where

V = volume in ml of standard hydrochloric acid solution used,

N = normality of standard hydrochloric acid solution, and

M = mass in g of sample of resin taken for the test.

6. DETERMINATION OF ACID VALUE

6.0 Outline of the Method — The acid value is the number of milligrams of potassium hydroxide necessary to neutralize the free acid groups of the polyamide. This is done by a titration of the resin solution in a solvent system.

6.1 Reagents

6.1.1 *Solvent Mixture* — Mix together toluene and butanol in 1:1 (*v/v)* ratio.

6.1.2 *Standard Alcoholic Potassium Hydroxide Solution* — 0.1 N in ethyl alcohol.

6.1.3 *Phenolphthalein* — 0.1 percent in ethyl alcohol.

6.1.4 *Procedure*

6.1.4.1 Dissolve 3-4 g of polyamide sample in a clean conical flask. Add 25-30 ml of the solvent mixture by gently warming. Titrate this solution against standard alcoholic potassium hydroxide using phenolphthalein as indicator. Express the result in milligrams of potassium hydroxide required per gram of sample.

6.1.5 *Calculation*

$$\text{Acid value} = \frac{V \times N \times 56{\cdot}1}{M}$$

where

V = volume in ml of standard potassium hydroxide solution used,

N = normality of standard potassium hydroxide solution, and

M = mass in g of sample of the resin taken for test.

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